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SPECIFICATION FOR NICOTINE SULPHATE SOLUTION

(First Revision)

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NEW DELHI 110002

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August 1984

Indian Standard

SPECIFICATION FOR NICOTINE SULPHATE SOLUTION

(*First Revision*)

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(*Continued on page* 8)

Indian Standard

SPECIFICATION FOR NICOTINE SULPHATE SOLUTION

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 29 June 1984, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Nicotine sulphate solution is extensively used in the control of insect pests of agricultural importance. It is manufactured from waste tobacco and from the liquors obtained from factories making chewing and smoking tobacco. The waste tobacco is macerated with water and lime, and then steam-distilled. The distillate is neutralized with sulphuric acid and concentrated.

0.3 This standard was first published in 1957. Subsequently one amendment was issued. This standard is being revised to make its requirements uptodate.

0.4 Nicotine sulphate solution is manufactured to contain 40 percent (*m/m*) of nicotine.

0.5 In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for nicotine sulphate solution.

2. REQUIREMENTS

2.1 Description — The material shall be a clear, yellowish brown to deep brown aqueous solution of the dibasic salt, nicotine sulphate, and may have acrid odour. It shall be free from sediment, scum, sludge, suspended matter or other extraneous impurities.

2.2 Nicotine Content — The material shall contain not less than 40·0 percent by mass of nicotine when tested by the method prescribed in Appendix A.

2.3 Sedimentation — The material shall satisfy the requirement of the sedimentation test prescribed in Appendix B.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as per requirements given in IS : 8190 (Part 2)-1980*.

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to any other information required under *Insecticides Act* and Rules:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;
e) Net mass of contents;
f) Nicotine content, percent (*m*/*m*); and
g) The cautionary notice worded as in the *Insecticides Act* and Rules.

3.2.1. Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

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*Requirements for packing of pesticides : Part 2 Liquid pesticides (*first revision*).

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in IS : 10627-1983*.

5. TESTS

5.1 Tests shall be carried out by appropriate methods referred to in **2.2** and **2.3**.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Clause* 2.2)

DETERMINATION OF NICOTINE CONTENT

A-1. METHOD

A-1.1 The method consists in steam distilling the nicotine from an alkaline medium, converting it into a salt by collecting the distillate in an acid medium, precipitating with silicotungstic acid and finally weighing the precipitate.

A-2. REAGENTS

A-2.1 Liquid Paraffin

A-2.2 Sodium Hydroxide Solution — aqueous, 40 percent (*m/v*).

A-2.3 Phenolphthalein Indicator Solution — 1·0 percent (*m/v*). Dissolve 1 g of phenolphthalein in 60 ml of rectified spirit and dilute to 100 ml with water.

A-2.4 Dilute Hydrochloric Acid — (1:4) and (1:1 000) by volume.

A-2.5 Silicotungstic Acid Solution — Dissolve 120 g of silicotungstic acid ($4H_2O.SiO_2.12WO_3.22H_2O$) in water and dilute to one litre. The solution should be free from cloudiness and green in colour.

NOTE — The silicotungstic acid should be white or pale yellow crystals free from green colour. Of the several silicotungstic acids, $4H_2O.SiO_2.10WO_3.3H_2O$ and $4H_2O.SiO_2.12WO_3.20H_2O$ do not give crystalline precipitates with nicotine and should not be used.

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*Methods for sampling of pesticidal formulations.

†Specification for water for general laboratory use (*second revision*).

A-2.6 Methyl Orange Indicator Solution — 0·04 percent (*m/v*), in aqueous ethyl alcohol (20 percent by volume).

A-3. PROCEDURE

A-3.1 Weigh accurately 0·5 to 1·0 g of the material in a suitable tared weighing tube. Quantitatively transfer the weighed material to a 500 ml Kjeldahl flask using water to wash the last traces of the material into the flask. If necessary, add to the contents of the Kjeldahl flask a little of liquid paraffin to prevent frothing during distillation, add a few small pieces of pumice to prevent bumping. Make the contents of the flask alkaline by adding a slight excess of sodium hydroxide solution, using phenolphthalein as indicator. Fit the mouth of the Kjeldahl flask with a two-holed rubber stopper through which passes the stem of a trap bulb and an inlet tube for steam. Connect the free end of the trap bulb to a well cooled condenser, the lower end of which dips below the surface of 10 ml of dilute hydrochloric acid (1:4) contained in a suitable receiving flask. Connect the inlet tube to the source of steam and distil rapidly with the current of steam. When the distillation is well under way, heat the Kjeldahl flask using a Bunsen burner to reduce the volume of the contents of the flask as far as practicable without causing bumping or undue separation of insoluble matter. Continue the distillation until a small quantity of the distillate shows no cloudiness or opalescence when treated with a drop of silicotungstic acid solution and a drop of dilute hydrochloric acid (1:4). Confirm the alkalinity of the residue in the Kjeldahl flask with phenolphthalein indicator solution. Reduce the volume of the distillate by concentrating it on a steam bath (*see* Note) and make up the volume of the concentrating distillate to a convenient volume in a volumetric flask with water at room temperature. Thoroughly mix the contents of the volumetric flask and filter through a dry filter paper, if it is not clear. Collect the filtrate in a convenient flask. Test a portion of this filtrate with methyl orange indicator solution to confirm its acidity.

NOTE — By heating on a steam bath the nicotine content of the distillate is not affected.

A-3.2 Pipette an aliquot (*see* Note) of the filtrate, containing about 0·1 g of nicotine, into a beaker and add at the rate of 3 ml of dilute hydrochloric acid (1:4) for each 100 ml of the aliquot and one millilitre of silicotungstic acid solution for every 0·01 g of nicotine supposed to be present in the aliquot. Stir the contents of the beaker thoroughly and let stand overnight at room temperature. Before filtering, stir the contents of the beaker to see that the precipitate settles down quickly and is in a crystalline form. Filter the contents of the beaker through an ashless filter paper. Wash all the residue remaining in the beaker on to the filter using dilute hydrochloric acid (1:1 000) at room temperature. Wash the filter with dilute hydrochloric acid (1:1 000) until a few millilitres of the filtrate from the tail of the funnel do not produce a precipitate or opalescence when tested with a few drops of the distillate containing nicotine (*see* **A-3.1**). Transfer the filter paper containing the precipitate to a tared platinium crucible, dry carefully and ignite the filter paper until all carbon is oxidized. Finally heat the platinum crucible over a Meker burner for not more than 10 minutes. Cool the crucible in a desiccator and weigh.

NOTE — If the nicotine content of the material is very low, an aliquot containing at least 0·01 g of nicotine should be used.

A-4. CALCULATION

A-4.1 Nicotine content of the material, percent by mass $= \frac{11 \cdot 41\ mV}{M \times v}$

where

m = mass, in g, of the residue (*see* **A-3.2**);

V = total volume, in ml, of the concentrated distillate after making up at room temperature (*see* **A-3.1**);

M = mass, in g, of the material taken for steam distillation (*see* **A-3.1**); and

v = volume, in ml, of the aliquot of the filtrate taken for the precipitation (*see* **A-3.2**).

APPENDIX B

(*Clause* 2.3)

SEDIMENTATION TEST

B-1. PROCEDURE

B-1.1 Transfer 10 ml of the material to a 100 ml conical flask. Add to it 50 ml of water and mix the contents of the flask by vigorously shaking for about five minutes. Allow the flask to stand at rest for one hour and examine for any sediment at the bottom of the flask.

B-2 REPORT

B-2.1 The material shall be deemed to have satisfied the requirement of the test if there is no sign of any sediment at the bottom of the conical flask.

(*Continued from page* 2)

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Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbo*	*Definition*
Force	newton	N	$1\ \text{N} = 1\ \text{kg.m/s}^2$
Energy	joule	J	$1\ \text{J} = 1\ \text{N.m}$
Power	watt	W	$1\ \text{W} = 1\ \text{J/s}$
Flux	weber	Wb	$1\ \text{Wb} = 1\ \text{V.s}$
Flux density	tesla	T	$1\ \text{T} = 1\ \text{Wb/m}^2$
Frequency	hertz	Hz	$1\ \text{Hz} = 1\ \text{c/s}\ (\text{s}^{-1})$
Electric conductance	siemens	S	$1\ \text{S} = 1\ \text{A/V}$
Electromotive force	volt	V	$1\ \text{V} = 1\ \text{W/A}$
Pressure, stress	pascal	Pa	$1\ \text{Pa} = 1\ \text{N/m}^2$

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AMENDMENT NO. 1 MAY 1994
TO
IS 1055 : 1984 SPECIFICATION FOR NICOTINE SULPHATE SOLUTION

(First Revision)

(*Page* 5, *clause* **4.1**) — Substitute the following for the existing:

'When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1983 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627 : 1983. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause **2.2** of the standard.'

(FAD 1)

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